

# Characterization of Ambient Air PM<sub>2.5</sub> in the Pittsburgh Region

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## Summary

PM<sub>2.5</sub> particle speciation analyses were performed on 24 hour filter samples, seasonally collected during the time period from spring 2000 through winter 2001. Selected filters were analyzed from the U.S. Department of Energy National Energy Technology Laboratory (NETL) Ambient Air Monitoring research sampling station. This research site is located in Southwestern Pennsylvania, approximately 15 km south of downtown Pittsburgh (40.30655° N latitude, 79.9794° W longitude, and elevation 325 m above sea level). The wind at this site is predominantly from the south and southwest.

Analysis methods used in this study included scanning electron microscopy / energy dispersive X-ray spectroscopy (SEM/EDX) for direct identification of particle species, ion chromatography (IC) for bulk

water soluble species, proton induced X-ray emission spectroscopy (PIXE) for bulk elemental analysis, and gravimetric measurements for total PM<sub>2.5</sub> mass. Microscopic analysis information was compared with and adjusted using data from bulk speciation and bulk gravimetric analyses in a complimentary fashion, in order to obtain a more complete accounting for all of the chemical / physical species that constituted the measured PM<sub>2.5</sub> material.

Scanning electron microscopy (SEM) was used in this study because it provides detailed information on the size, morphology and elemental composition of individual particles. This information provides a classification of sample particle constituents, and possible source association information that is not available from many of the commonly used bulk analytical methods (Casuccio et al, 1983). Data from the SEM techniques complemented traditional analytical data obtained from ion chromatography (IC) and proton induced x-ray emission (PIXE). SEM data was essential to identify spherical aluminosilicate (SAS) particles that can be used as a tracer for primary emissions from pulverized coal fired electric power generation stations (Eatough et al, 1997).

Samples for SEM analysis were collected at the NETL site, using a four channel Andersen RAAS<sup>®</sup> 400-PM<sub>2.5</sub> speciation sampler. Each sample was collected over a 24-hour time period from noon to noon. During each sampling experiment, one standard Federal Reference Method (FRM) Teflon filter was collected in a R&P 2025 Sequential Sampler.

Primary and secondary particles were counted from samples collected on polycarbonate (PC) filters during this time period. The filters were pre-coated with palladium to enhance the secondary electron image quality and improve identification of carbonaceous particles by providing an internal standard EDS signal threshold value.

The PC filters were prepared for SEM analyses by cutting a wedge from the filters and mounting it on a substrate with double-sided adhesive silver tape. Images and elemental spectra were collected on ~1200 particles per sample using CCSEM procedures (Personal SEM, Aspex Instruments, LLC). The SEM was run in secondary electron imaging mode to characterize particles down to 0.16µm diameter.

SEM parameters included a 15 kV accelerating voltage, a sample working distance of 16 mm, 7 seconds EDX spectra acquisition for each particle, and multiple beam current checks with an internal Faraday cup during each filter section analysis. Consistency adjustments to the image threshold gray level values set for particle recognition were periodically made to compensate for small variations in the image brightness and contrast due to sample charging effects. Magnification and X-ray calibrations were also incorporated into each analysis. EDX spectra, collected at 15 kV on traceable reference standards were used in developing a spectral library for reference during the analyses.

To correctly identify carbonaceous particles during each CCSEM filter analysis, the carbon to palladium ratio (C:Pd) was measured on an individual filter basis, by collecting multiple point particle free filter background spectra on each filter sample. These EDX ratio values were incorporated into the individual filter analysis CCSEM rule set to provide an accurate carbonaceous particle classification 'on line' during each filter analysis.

Based on data collected from autumn 1999 through the present, it has been determined that PM<sub>2.5</sub> at the NETL site consists of three primary components: sulfate (assumed ammonium sulfate), carbonaceous material, and 'crustal' material.

All of the 24-hour filter data, and the carbon monitor data was normalized to a  $\mu\text{g}/\text{m}^3$  basis in order to directly compare the information obtained by different analytical methods. The SEM data were calculated with the assumptions that the analyses accounted for all of the particulate material volume on the filters. Since the PC filter loading closely agreed with the FRM filter loading on any given day, normalized for the actual volume flow through the filters (Martello et al, 2001), this assumption was reasonable.

It was found in this study that the SAS constituent comprised a very small fraction of the total material collected. It was also found that a portion of the carbonaceous material was characteristic of emissions from mobile sources. Comparison of other particle types with source sampling can help to determine other generic sources of the material collected in this study, in order to more completely define and account for the total PM<sub>2.5</sub> mass.

## References

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